FINAL TECHNICAL REPORT

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GRANT TITLE: The Effects of Physical Composite Particle Parameters on the Efficacy of a Proprietary Sound-Attenuation Technology and Its Application to Advanced Structural Composites

AWARD PERIOD: April 1, 2000 – December 31, 2002

OBJECTIVE: Two principal objectives were proposed in this research: (1) determine the acoustical effects of the filler materials with different sound impedance in composites fabricated according to a proprietary technology, and (2) determine the feasibility of applying the proprietary technology to continuous fiber-reinforced advanced structural composites.

APPROACH: The sequential experimental approach began by attempting to apply the acoustical technology with neat epoxies using off-the-shelf filler materials possessing a range of physical characteristics. The selected filler materials were successfully used in rubber-type materials to achieve acoustic attenuation properties in the past. Secondly, filler materials with carefully controlled characteristics were introduced into the composite resin matrix formulas and their influences on processing property, material microstructure, acoustic attenuation and final mechanical properties were studied and recorded. A novel processing technique was developed to accomplish good filler distribution in continuous fiber-reinforced composites with 50% V_f, based on experimental studies and microstructure observation via microscopes. A DOE (design for experiment) approach was used to explore filler type and interactions on the acoustic, damping and mechanical properties of the resulting composites.

ACCOMPLISHMENTS (throughout award period): Two filler materials were selected and used in this research based on NAMRL (Naval Aerospace Medical Research Laboratory) previous acoustic attenuation results: SPHERICEL 110P8 Hollow Glass Spheres (Potters Industries, Inc.) and the Iron Powder FE-101 (Atlantic Equipment Engineers – High Purity Metals and Compounds). The SPHERICEL is a hollow glass bead (spherical shape) with an average size of 11.7 μm. The FE-101 is a 99.9% pure iron compound, which is rough and irregular. These particles were screened with a 5 μm sieve. However, as with any sieving process, this only controlled the size of the particles in two dimensions; the resulting third dimension of any of the particles could exceed 5 μm. The actual shape of the iron powder was more whisker shaped with certain aspect ratios. The fiber reinforcement used in the research was a bi-directional warp-knitted glass-fiber, COFAB A1118 / 0-90 BIAXIAL (CollinsCraft Composites).

Due to its affordability and good dimensional accuracy, the vacuum assisted resin transfer molding (VARTM) process was initially selected to produce composite panels by adding single or mixed types of the fillers at different loading level. Epon 862/EPI CURE W (Shell Chemicals) resin matrix was used. The influences on the viscosity of the resin matrix by varying the temperature and adding the fillers are shown in Figure 1. Based on the viscosity analysis and experimental results, the resin and mold temperature was set at 120°C to ensure low viscosity and complete mold filling during resin infusion. At this temperature, the gel time or processing window of the resin matrix was more than one hour. The composite panels of 44v% fiber load and different filler contents were produced. However, two major issues occurred for these panels. One issue was the filtration of the particles by the fiber networks during long distance resin flow of mold filling for the VARTM process. The coloration gradients that can be seen on the surface of some panels, as shown in Figure 2, were the results of the filtration of the filler particles by the dense fiber network. Another issue was accumulative distribution of the powder fillers in the composites. A further microscopic analysis was conducted on polished sections of the resulting samples. A representative picture

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of the composites with iron powder fillers is presented in Figure 3. An image of the composites mixing with both iron and glass fillers is provided in Figure 4. The accumulative distribution and separation of the powder fillers occurred due to two major reasons. Density differences between the resin and filler obviously were mainly responsible for this uneven distribution. The densities of resin, iron and glass fillers were 1.3g/cm^3, 7.23g/cm^3 and 1.13g/cm^3 respectively. The relatively low infusion viscosity (less than 100cP) and long gel time (more than 1 hour) also provided the opportunities to let light glass filler float and high iron particle sink. This uneven particle distribution is not acceptable for acoustic attenuation applications.

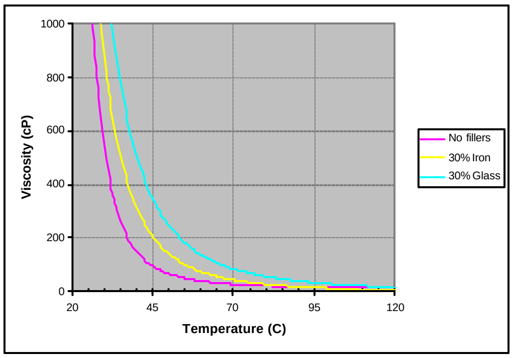

Figure 1 Epon 862/EPI CURE W resin matrix rheology with and without powder fillers

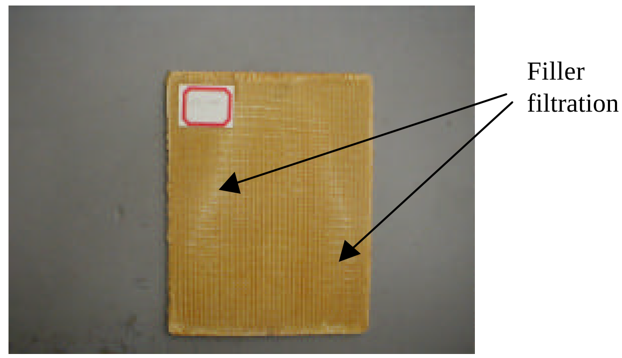

Figure 2 Filtration of the fillers during VARTM processing

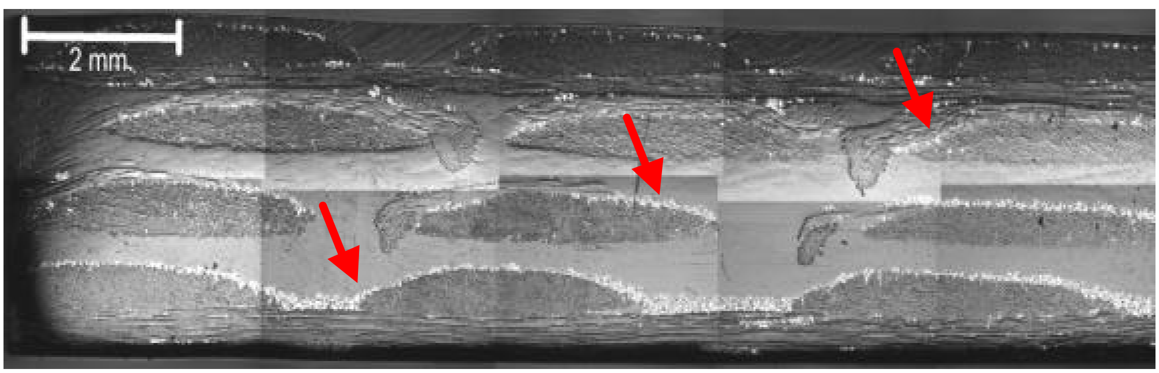

Figure 3 Cross-section of the composites with iron filler (bright particles indicated by the arrows) of VARTM process

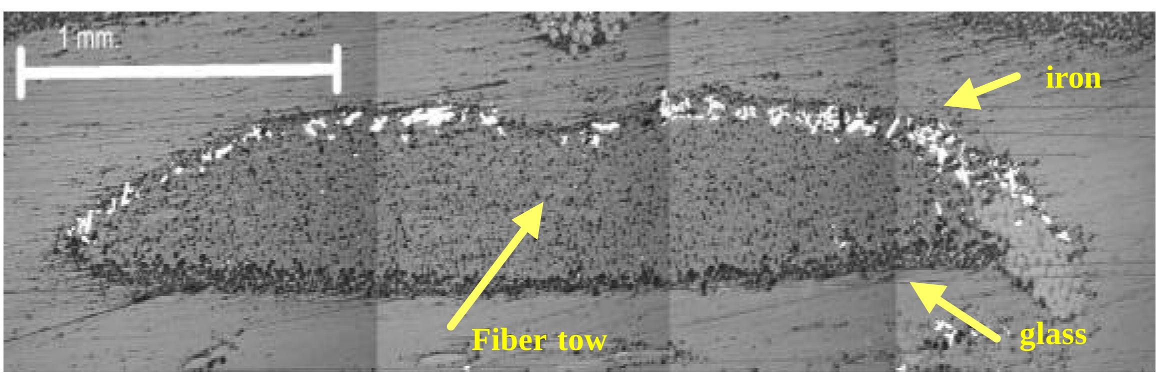

Figure 4 Cross-section of the composites with mixing both iron (bright particles) and glass filler (dark particles) of VARTM process

To solve processing issues of filler filtration and accumulative distribution, a novel process technique of hand lay-up/rotating molding was developed. By using hand lay-up process instead of the VARTM process, the travel distance of the resin/filler mixtures can be largely reduced and almost eliminate the particle filtration phenomenon. A new resin system, WEST SYSTEM 105 Epoxy Resin and WEST SYSTEM 205 Fast Hardener (Gougeon Brothers, Inc.), was also investigated and used for the process. The viscosity of this new system was greater than 600cP, with a gel time much less than 1 hour at curing temperature. The new resin system was able to partially reduce the gravity effects of the fillers. Another main modification was also made to solve the gravity problem. This modification consisted in rotating the mold while the resin was gelling in order to improve particle distribution uniformity in the thickness direction. After compressing the resin, the mold was clamped onto a mold-rotating device, as shown in Figure 5. The mold was then rotated 180° every minute for one hour after the mold was closed and began to cure. The microstructures of the composite panels using hand lay-up/rotation molding are shown in Figure 6 and 7. Both filler particles were more uniformly distributed in the resin region and partial penetrated into fiber tow. No observable accumulative distribution resulted due to gravity effects as seen in previous products of VARTM process. This new process technique was used for the composite fabrication in the research.

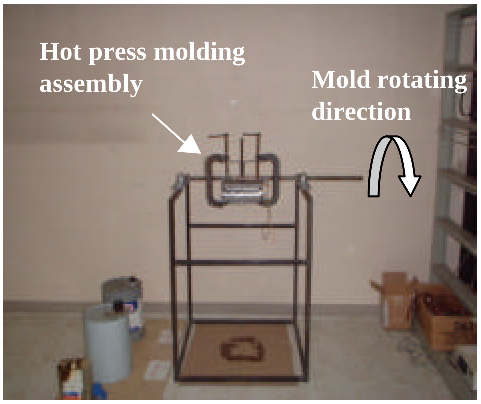

Figure 5 Assembly of rotation hot press molding

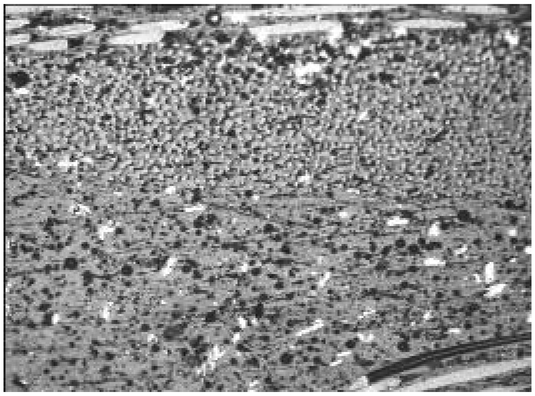

Figure 6 Uniform distribution of iron (bright irregular shapes) and glass (black spots) fillers

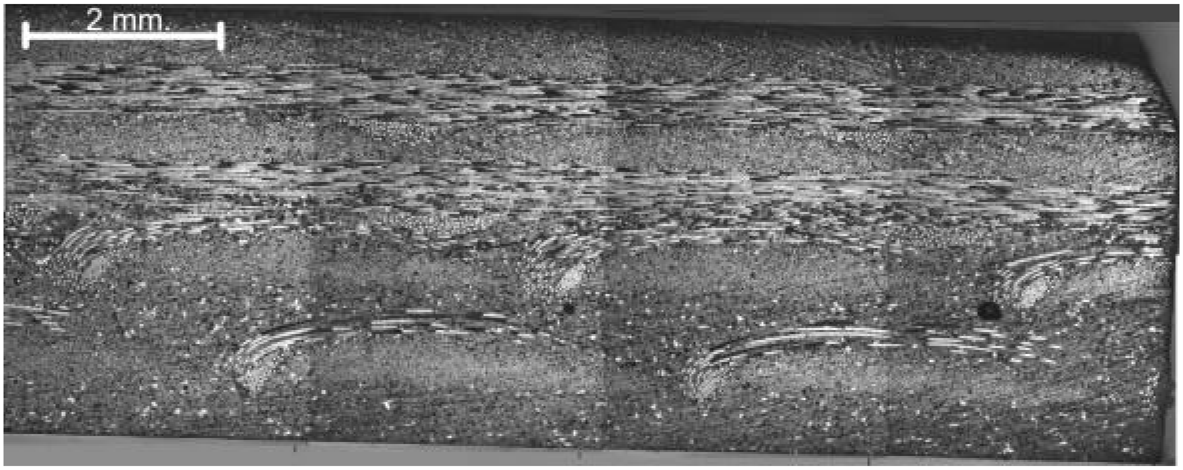

Figure 7 Microstructure of the composites with both iron (bright irregular shapes) and glass (black spots) fillers (50v% fiber loading)

To efficiently identify the filler influences on acoustic, damping and mechanical properties of the continuous fiber-reinforced composites, a modified design of experiment (DOE) approach was used in the research. The modified 2^2 factorial DOE used in the research is shown in Figure 8. All the composites

fabricated based on this DOE design had 50% fiber volume content. Acoustic attenuation measurements of the composites were taken in a semi-reverberant sound chamber using a pink noise source to produce a uniform sound pressure field of 120 dB (SPL) at NAMRL. The acoustic attenuation at low frequency (less than 500Hz) was our particular focus for this research. Initially acoustic testing showed that the original sample fixing procedure developed for rubber-like materials was not suitable for high stiffness structural composites. A modification of the fixing procedure was conducted, in which the composite samples were positioned flat so as to cover a sensing microphone mounted in the high mass flat plate coupler. The sealing between the composite sample and the coupler was achieved by applying vacuum grease and clamping mass. The acoustic attenuation results are given in Figure 9. All composite samples of 1mm thickness with and without the fillers have reached the maximum test range of acoustic attenuation for the experiment setup at NAMRL, which is shown in Figure 9 as a solid dark line. The composites demonstrate very high Transmission Loss (TL) or acoustic attenuation at low frequency range. For example, the composite sample offered 40-50 dB TL at 33.6 Hz, compared to 10-30 dB TL for rubber materials filled with sample particles. However, due to the limitation of the experimental setup, the detail influences of the type, concentration and mixing ratio of the fillers have not yet been identified. Further improvements in the acoustic test setup are required.

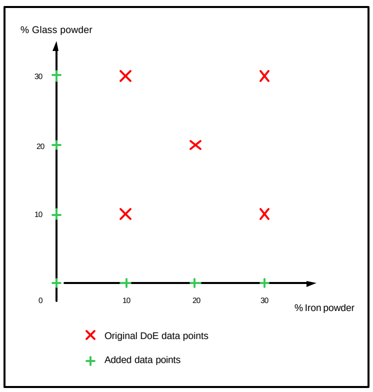

Figure 8 DOE design for composite fabrication and Property investigation

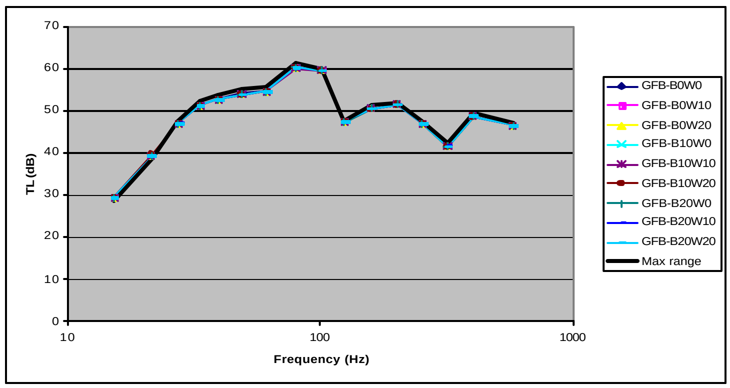

Figure 9 Transmission Loss (TL) of the composites (B20W20: mixing 20wt% iron fillers and 20wt% glass fillers in the resin)

Additional dynamic mechanical analysis (DMA) tests were also carried out to further explore the filler influences on energy absorption natures of the composites. The DMA results provided some information on the vibration properties that the acoustic measurements were unable to provide. Around frequencies of approximately 50 Hz, we could see a very strong contrast damping behaviors between the different composite samples, depending on whether or not they contain particles, which is a good indication of the filler influences on damping or possible acoustic properties. The mechanisms of the different damping behaviors of the composites are not yet clear.

Tensile tests according to ASTM D3039M standard were also performed using statistical techniques for design and analysis of the filler influence on mechanical properties of the composites. The ANOVA analysis of testing results, based on the DOE design, revealed that the particles do not have a significant impact on the tensile modulus but have a significant effect on the tensile strength properties, compared to the composites without any fillers and having the same fiber volume content. Furthermore, this effect seemed to be very dependent on the geometry of the particles since the different powder types had opposite effects. The results show that spherical shape glass filler could slightly decrease tensile strength. However, the whisker shape iron filler could apparently increase composite tensile strength.

CONCLUSIONS: A new composite processing technique, hand lay-up/rotation molding, was successfully developed to fabricate continuous fiber-reinforced composites with the selected iron and glass powder fillers for acoustic attenuation. The microstructure analysis of the resulting composite samples show that the developed process can achieve uniform filler particle distributions for both fillers with different particle size and gravity. The results of acoustic transmission tests show that all fabricated composite samples of a

thickness of 1mm with or without the filler resulted in a transmission loss equal or higher than the maximum test range of the testing setup at NAMRL. Further DMA analysis indicated the filler influences on material damping property experimentally. The statistic analysis also revealed that the particles do not have a significant impact on the tensile modulus but have a significant effect on the tensile strength properties, comparing to the composites without any fillers and having the same fiber volume content. This influence is dependent on the geometry of the selected fillers.

SIGNIFICANCE: The hand lay-up/rotation molding process developed in the research is capable to achieve uniform distribution of the selected fillers for acoustic attenuation in continuous fiber-reinforced structural composites. The acoustic test results show that the resulting composites with a thickness of 1 mm can offer a high transmission loss at low frequency range, compared to rubber/filler acoustic attenuation materials. The influences of the fillers on composite tensile property were also investigated. The study further proved the feasibility of development of structural composites with acoustic attenuation feature.

PATENT INFORMATION: None

(The investigators have discussed with NAMRL researchers concerning applying for patent protection for several of the modified manufacturing techniques and for the resulting material formulas. After further research both parties anticipate applying for patents.)

AWARD INFORMATION: None

REFEREED PUBLICATIONS (for total award period): None

(Because the Navy's technology has been licensed by commercial concerns and several companies are engaged in cooperative research and development agreements with an eye towards commercialization of the technology, legal concerns strictly limit the degree to which findings derived from this research can be disseminated. As a result, no open literature or other publications have been issued.)

BOOK CHAPTERS, SUBMISSIONS, ABSTRACTS AND OTHER PUBLICATIONS (for total award period): None

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